CEMENT AND LIME MANUFACTURE

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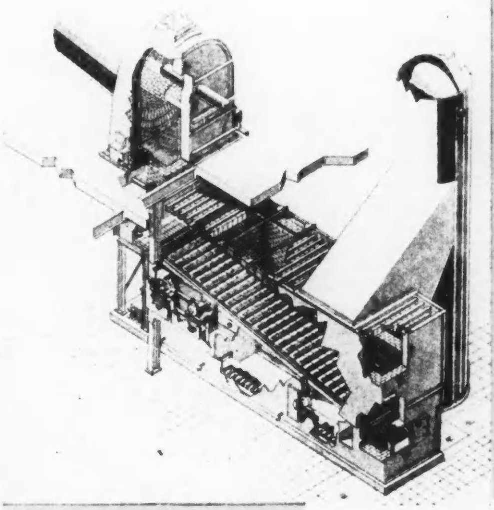

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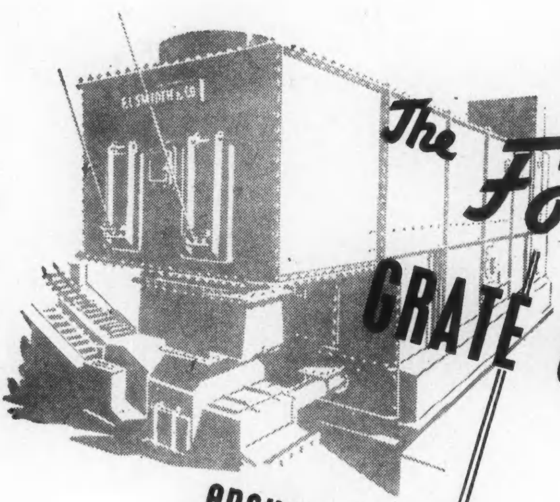

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Vertical Cement Kilns.

THE modern vertical cement kiln has many new features, such as increased capacity, reduced fuel consumption, and uniform quality of clinker. A shaft kiln installation designed for a daily output of 150 metric tons of clinker occupies only 650 sq. ft. of floor space. Another advantage of the shaft kiln is that its operation can be stopped at any time with little trouble.

A low shaft kiln, designed by W. Anselm, of Germany, is shown diagrammatically in *Fig.* 1. Its main features are the introduction of combustion air in the centre of the charge and the cooling of the clinker in a rapid grate-type cooler. The air required for combustion is supplied under pressure through two pipes, one of which enters the charge at the bottom so that the incoming air helps to reduce the temperature of the clinker before it leaves the kiln. The other pipe

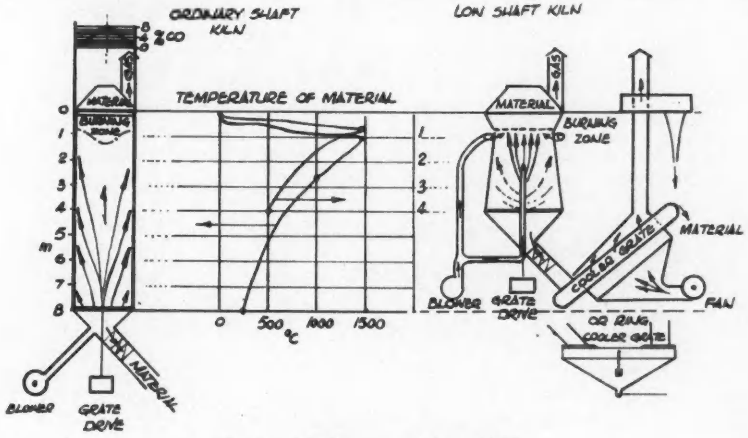

Fig. 1. Ordinary and Low Kilns.

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supplies air directly to the burning zone. The temperature of the clinker discharged from the kiln is about 500 deg. C. The clinker is then further cooled on the grate, from which it is discharged at a temperature of about 100 deg. C. A feature of the kiln is the small amount of dust it produces, which is less than 0·5 gramme per cubic metre. The overall thermal efficiency of the kiln is about 45·7 per cent. This kiln, with a diameter of 3·2 metres, has a capacity of about 225 tons of clinker per day.

A Gas-Fired Kiln.

Another kiln, by the same designer, is adapted to use oxygen-enriched air for combustion. The kiln-grate and discharge-gates are power operated, and the kiln may have a daily capacity of about 300 tons of clinker. Instead of using air for combustion a mixture of gases is prepared containing any desired ratio of oxygen to carbon dioxide. The waste gas from the kiln is used for this purpose after it has been cleaned in a wet-gas washer and the dust has been removed in

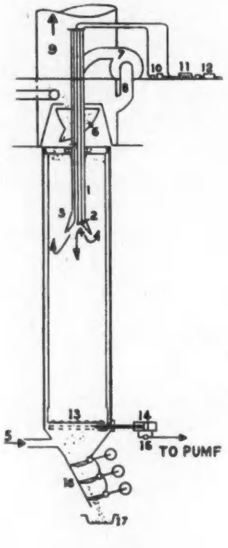

Fig. 2.

1, Chute; 2, Oil-burner; 3, Water Pipes; 4, Vaporizing chamber; 5, Secondary air inlet; 6, Rotating feeder for pellets; 7, Primary air fan; 8, Flue gaspipe; 9, Chimney; 10, Pump; 11, Heat exchanger (oil-water); 12, Oil pump; 13, Discharge grate; 14, Cylinder; 15, Valve; 16, Discharge gates; 17, Conveyor.

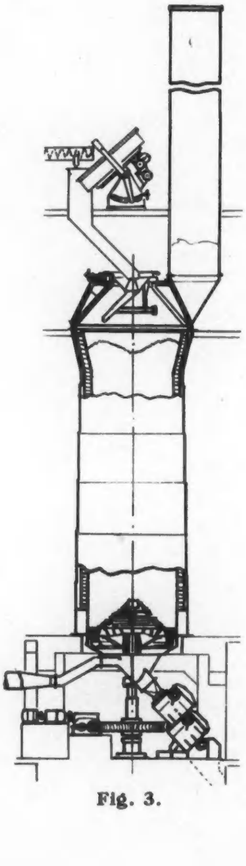

Fig. 3.

cyclones. The capacity of the kiln per cubic metre is up to 6 tons in 24 hours, so that with a height of 10 metres and an internal diameter of 2·5 metres the capacity of a single shaft kiln is equal to that of a rotary kiln 60 metres long.

Tests show that the most suitable mixture of gas is about 40 per cent. oxygen, 40 per cent. carbon dioxide, and 20 per cent. hydrogen. The oxygen used is "tonnage oxygen," produced on a large scale, which contains about 80 per cent. of pure oxygen. The kiln is suitable for fully-automatic operation. Heat consumption, when the proportion of oxygen in the combustion air is 20·9 per cent., is 1100 k./cal. per kg. of clinker when the output is 110 tons per 24 hours. When the amount of oxygen in the combustion air is 40 per cent. the heat consumption is 800 k./cal. per kg. of clinker and the kiln output is 215 tons in 24 hours.

Fig. 4. Weighing Equipment.

Against the advantages of reduced fuel costs, increased output, and improvement in the quality of clinker, there should be set the cost of producing oxygen.

Oil-Fired Kilns.

A diagram of an oil-fired shaft kiln designed by W. Anselm is shown in *Fig.* 2. Only a few kilns of this type are in use; no operating data are available, but development work is being undertaken in several countries as the use of fuel oil is expected to be advantageous.

Coal-Fired Kilns.

In the past few years several shaft kilns made by L. de Roll, S.A. of Zurich, have been installed in various countries. A battery of four such kilns, with pneumatic homogenizing installation and ancillary equipment, has recently been ordered for an American cement works. *Fig.* 3 is a diagram of this type of kiln.

Fig. 5. Rear view of Pelletisers.

Fig. 6. Pelletiser.

The coke or anthracite-breeze and the raw meal are first weighed and proportioned on automatic scales (*Fig.* 4) and mixed in a paddle-mixer to a homogeneous mass, which is passed on to a pan-pelletiser where it is formed into nodules, from 10 to 20 mm. diameter, by adding 12 to 14 per cent. of water. The nodules leaving the pelletiser are fed directly to the kiln. The pelletiser (*Figs.* 5 and 6) is an inclined rotating pan in which the water is added to the mixture and nodules of a uniform size are formed. By adjusting the inclination of the pan and its speed of rotation, the size of the nodules can be varied. The nodules are strong enough to support the weight of the kiln charge without being crushed, so that the gases can circulate freely in the burning zone.

Fig. 7. Discharge Gate and Air Lock.

Coke and anthracite are recommended as the fuel because they contain very little volatile matter and there are no losses due to volatile constituents being distilled off before reaching the burning zone, which would lower the overall efficiency of combustion. The calorific value of the fuel should be as high as possible, preferably 11,700 to 12,600 B.T.U. per lb. and not less than 9900 B.T.U. per lb. (coke having 22 per cent. ash and 10 per cent. moisture content is suitable). The anthracite should contain 6 to 8 per cent. volatile matter, 6 to 15 per cent. ash, and 4 to 6 per cent. moisture; the coke should contain 2 to 4 per cent. volatile matter, 6 to 15 per cent. ash, and 4 to 16 per cent. moisture. The particle size of coke or anthracite coal should not be larger than $\frac{5}{16}$ in., and the proportion smaller than $\frac{3}{64}$ in. should be as low as possible; as much as possible should be

between $\frac{3}{64}$ in. and $\frac{1}{4}$ in. Extremely fine particles increase the quantity of carbon monoxide in the exit gases. A high proportion of particles larger than $\frac{1}{4}$ in. requires an unnecessary increase in the length of the burning zone and results in the clinker being discharged at a higher temperature. The size of the fuel can therefore have a considerable effect on the heat produced from a given quantity of fuel.

The raw material should be ground as finely as practicable, as the finer it is the better the quality of the clinker. The fineness is limited by the high cost of very fine grinding; it is therefore recommended that the raw meal be ground to a residue of 10 to 15 per cent. on B.S. sieve No. 170.

The kiln is charged from a hopper with an adjustable spout. The nodules are distributed uniformly in the kiln and the kiln is kept charged to a constant level and operates continuously. In the enlarged upper part the material is first dried and then falls slowly to the narrower part. Decarbonation takes place at about 950 deg. and the final burning of the clinker at 1450 deg. C. The nodules keep their shape very well as they pass through the kiln, thus helping to maintain a uniform distribution of gas throughout the whole cross section, and the burning zone stays at a fixed level. The clinker is cooled in the lower part of the kiln before it reaches the discharging grate.

The clinker is discharged through a rotary grate and a gate with a triple air lock (*Fig.* 7). By adjusting the speed of rotation of the grate the amount of clinker discharged, and thus the rate of burning, can be controlled. The average fuel consumption stated by the manufacturers is 950 to 1,000 k./cal. per kg. of clinker with a power consumption of 12 to 14 kWh. per ton for the whole installation, including the blower.

Congress of Chemical Engineering 1958.

The European Congress of Chemical Engineering 1958 is to be held in conjunction with the ACHEMA Congress at Frankfurt-am-Main from May 31 to June 8, 1958. Further information can be obtained from DECHEMA, Frankfurt-am-Main 7, Postfach, Germany.

Cement Works in Peru.

A contract valued at 87,000,000 soles has been placed with a Danish firm for the supply of machinery and plant for a new cement works to be built at Caracoto, in southern Peru. The factory will have a capacity of about 60,000 tons a year.

The Cement Industry in Ceylon.

The output of the cement works at Kankesanturaia in the year ended September 30, 1956, was 87,000 tons, an increase of 10,000 tons on the previous year.

Cement Production in Finland.

The production of Portland cement in Finland in 1956 exceeded a million tons. This is the highest production on record.

Hardening of Cement in Cold Weather.

ENGINEERS from many countries where concreting has to be carried on at low temperatures attended a symposium on Winter Concreting held in Copenhagen last year under the auspices of the Danish National Institute of Building Research, and presented the results of their experience in overcoming this problem. Research workers also attended and described the results of laboratory investigations on the effect of low temperatures on the properties and rate of hardening of cements. These papers have now been published in a volume* of 1574 pages, which includes the contributions to the discussions which followed the papers. The volume is printed in the English language, and includes an extensive summary of the main conclusions in English, French, and German. Among the papers presented and contributions to the discussions published in this volume are.

WEATHER IN RELATION TO WINTER CONCRETING: Temperature-variations in Sweden in relation to tests of resistance to freezing, by Hans A. Vinberg. Calculation of the influence of the weather on concrete, by Jørn Jessing. Winter concreting in Poland, by M. Rzędowski. Contributions by J. G. Buitink, F. N. Sparkes, and E. G. Suenson.

LABORATORY EXPERIMENTS ON THE DETERMINATION OF THE RESISTANCE OF CONCRETE TO EARLY FREEZING: General Report, by Inge Lyse. Resistance to early front action, by Göran Möller. Cold-weather concreting with high-early-strength cement, by Ernst Gruenwald. Effect of freezing on strength and expansion, by Arvo Nykänen. Tests of concrete at a temperature of 20 deg. F. (– 6·7 deg. C.), and Effect of entrained air and calcium chloride, by Lewis H. Tuthill. Freezing and thawing tests on green concrete, by Poul Nerenst and Niels Munk Plum. The influence of alcohol on concrete in cold weather, by Erik V. Meyer. Contributions by G. Möller, P. Lhopitallier, G. Wastlund, T. C. Powers, K. Schaden, A. Klein, and I. Lyse.

EFFECT OF TEMPERATURE ON THE HARDENING OF CONCRETE: General Report by the Swedish Cement and Concrete Institute. Curing by electric heating, by K. Horimatsu. Computation of times for removing formwork, by Hans A. Vinberg. Effect of temperature on the heat of hydration, by E. Rastrup. Hardening at different temperatures, especially below freezing point, by Arvo Nykänen. Effect of initial curing on strength, by T. Takahashi and M. Hayashi. Effect of low temperature curing on compressive strength, by J. D. McIntosh. Hardening at different temperatures, by C. J. Bernhardt. The heat of hydration of cement, by U. Danielsson. Winter concreting in the Soviet Union, by S. A. Mironov. The use of the "Maturity"-function in assessing the sensibility of cements to low temperatures, by W. Brand. Hydration of cement as a function of temperature, by E. Rastrup. Contributions by E. Rastrup, P. Håkansson, W. Czernin, S. G. Bergström, T. N. W. Akroyd, M. Rzędowski, E. Lewicki, A. Klein, H. Granholm, and G. Wästlund.

* "RILEM Symposium on Winter Concreting". Obtainable from Concrete Publications, Ltd. Price 65*s.* (by post 67*s.*); $12.50 in North America.

RESISTANCE TO FROST AT EARLY AGES: General Report, by T. C. Powers. Influence of the age of concrete before exposure to freezing on strengths beyond 21 days, by J. Blondel. Computation of resistance to freezing at early ages, by P. Nerenst. Effect of air entraining in cold weather, by V. V. Stolnikov. Evidences of disintegration of concrete affected by freezing and thawing, by Ervin Poulsen and G. M. Idorn. Contributions by T. C. Powers and W. Czernin.

ENSURING HIGH-QUALITY CONCRETE IN WINTER: General Report, by A. Voellmy. Television tower at Stuttgart—a steel-concrete structure built in cold weather, by E. Bachus. Influence of heated mixing water on properties of concrete and heat of hydration of cement, by Hideo Yokomichi. Electrical curing in cold weather, by Yasuo Ichiki. Setting temperatures at air-temperature below freezing, by Chr. F. Grøner. Temperatures maintained by insulation, by Lewis H. Tuthill. Classification of durability according to resistance to frost, by S. V. Shestoperov. Heat treatment by steam at atmospheric pressure, by K. Thiel. The use of chloride salts, by S. A. Mironov and B. A. Krylov. Contributions by I. Grzymek, M. Kohn, P. Haller, F. Scheidegger, Inge Lyse, K. Böhmer, W. Grün, A. Lazard, A. Voellmy, J. Jambor, H. Rüsch, J. D. McIntosh, F. Hess, and A. Staub.

CONSTRUCTION PROCEDURE IN COLD WEATHER: Winter concreting in Finland, by Beato Kelopuu. Winter concreting in Holland, by J. G. Buitink and J. M. L. Trouw. Construction of houses and multiple story buildings in cold weather, by Arvo Nykänen. Use of ready mixed concrete in winter, by Folmer Jørgensen. Winter concreting in China, by L. S. Wu. The hardening of concrete in frost, by V. N. Sizov. Electrical heating of massive structures such as dams, by K. V. Alexeyev. Contributions by H. Rühle, J. J. Bouvy, K. Schaden, J. Talbierski, A. Klein, and F. W. Katlein.

Research on Cement in France.

A conference is to be held in Paris on June 3 and 4 to commemorate the tenth anniversary of the foundation of the Centre d'Etudes et de Recherches de l'Industrie des Liants Hydrauliques. The meetings will be presided over by M. Albert Caquot, and papers will be presented by H. Lafuma (France), A. Allan Bates (U.S.A.), A. R. Collins (Great Britain), R. Dutron (Belgium), Fr. Keil (Germany), Donovan Werner (Sweden), W. Czernin (Austria), and Ed. Torroja (Spain). There will also be a visit to the laboratories of the Centre. Further information may be had from the Centre at 197 Boulevard Saint-Germain, Paris VII, France.

Production of Cement in Mexico.

The production of cement in Mexico in the year 1956 was 2,276,660 tons, compared with 2,085,652 tons in 1955.

Cement Production in Nicaragua.

The production of cement in Nicaragua in the year 1955 was 24,600 tons, compared with 22,500 tons in 1954.

Proposed International Standard for Testing Cement.

THE Swedish Cement Statistical and Technical Association, known as Cembureau, has for some years studied the question of promoting international standards for cement with the object of securing uniformity in requiiements throughout the world. The Association in 1949 appointed a "working party," on which twelve nations are represented. Most of the experimental work has been on tests for strength, but other properties have also been investigated. On many questions the party has arrived at more or less definite conclusions, but some problems are yet unsolved. The report gives information on what has been agreed upon, and the position of the work still in progress. The following are among the agreed recommendations.

Additions of other materials not exceeding 1 per cent. may be interground with Portland cement clinker at the option of the manufacturer, provided that such additions will not be harmful.

Supersulphated cement is the product obtained from a mixture of granulated blastfurnace slag, calcium sulphate, and a catalyst which may be lime, clinker, or cement, provided that (a) the proportion of SO_3 in the mixture exceeds 5 per cent., and (b) the proportion of catalyst in the mixture does not exceed 5 per cent.

As few restrictions as possible should be imposed on chemical composition, and no general rules should be laid down. A content in Portland cement of 5 per cent. MgO is considered safe in all cases. No decision has been reached on the allowable sulphate content, but a tentative figure of 3·5 per cent. has been suggested. Concrete generally shrinks when drying, and for that reason slight expansion due to sulphate action should not be harmful. No case was known to the committee where damage due to too high a proportion of sulphate had occurred.

The Le Chatelier test for soundness and the Vicat method for setting-time are considered to be satisfactory and are recommended.

It is not necessary to limit the fineness of cement. Nevertheless, it is advisable to have standard methods for assessing the fineness if that is required. For routine purposes the sieve residue should be determined as well as the specific surface. For determining specific surface, air-permeability methods are preferred, and the Lea-Nurse apparatus is recommended because it can easily be calibrated directly. The modification by Blaine is recommended for routine purposes; it is simpler to use and can be calibrated against the Lea-Nurse apparatus. The Andreasen method is best for assessing particle-size distribution, and is recommended for research work.

It would seem desirable to develop a method relating the strength of cement to the strength of concrete, that is a concrete test. This, however, requires more work than a mortar test, and it appears to be more difficult to standardize aggregates for concrete than for mortar. The committee concluded that in the first place a mortar test was most likely to be agreed upon and should be developed, and that a concrete test should be regarded as reference test.

Proposed Mortar Test.

The proposed mortar test is described in full, and an account is given of the tests made in different countries before deciding on this procedure. The main characteristics of the mortar are based on the following considerations. (1) Except in the case of special high-strength concrete made with a low water-cement ratio, concrete generally has relatively high water content, and has strengths and rates of hardening different from those of the "dry" mortars still in use in the specifications of many countries. (2) Plastic mortar has a rate of hardening more like that of concrete and the relation between the strength of concrete and the strength of mortar varies less with age for a plastic than for a "dry" mortar. Plastic mortar consequently is an advantage even though it may not permit an exact prediction of the strength of a concrete mixture. (3) The mortar should have sufficient plasticity for easy moulding, thus reducing the influence of the operator; it should also be able to retain water sufficiently to minimise "bleeding." These requirements could be met by a high cement-sand ratio in the mortar. The proportions 1 : 3 are generally used, however, and the committee considered that it does not favour a new departure. (4) In most countries the standard sand used has a narrow range of particle sizes, and this does not produce the required plasticity. In other countries the sands used are composed of several fractions of different sizes, and this latter type of sand has been accepted by the committee. (5) The committee has found it desirable to adopt a constant percentage of water in the mortar rather than to vary it according to the quantity required to give a cement paste of normal consistency. This quantity varies with different operators and, by fixing the amount of gauging water, a source of variation is avoided. (6) The composition of the mortar, its moulding, and the test to which it is submitted, should be such that the results obtained are consistent and reproducible. The committee recommends that the tensile test on 8-shaped specimens be discarded and replaced with a bending test. The size of the specimens proposed is that which Feret recommended long ago and which several countries have already adopted, that is 4-cm. by 4-cm. by 16-cm. prisms.

Tests on Concrete.

Tests on concrete have been made because they constitute the first step in developing a mortar test having the best correlation with concrete. The development of a concrete test suitable as an acceptance test in specifications had earlier not been considered practical, chiefly because of the difficulties anticipated in defining the nature of the aggregate. However, the Associated Portland Cement Manufacturers, Ltd., of Great Britain, suggested a concrete test in which any aggregate could be used provided that the amounts of cement and water in a specimen were exactly correct, the concrete was reasonably workable and had a slump between ½ in. and 2 in., and the aggregate had sufficiently high strength and low water absorption. This test is about to be introduced into the British Standard Specification for Cement. In its original form it was used by the Associ-

ated Portland Cement Manufacturers, Ltd., for internal use but not in a form suitable for a standard specification. The method was revised and tested by the committee in 1952, and it was further revised and retested in 1953. The results of these tests gave sufficient confidence to propose it as a British Standard. It was then tested by the British cement industry and two British Government laboratories. This was followed by further tests at more than twenty laboratories in England and one in South Africa. This is an example of a standard test produced by an international organisation being accepted in a national specification, and it is believed that it is the first time that such an action has been taken.

The report, entitled "On the Testing of Cement," is obtainable from Cembureau, P.O. Box 245, Malmö, Sweden (price 10s.).

Production of Cement in Turkey.

In the year 1956 the production of cement in Turkey was 970,000 tons and 291,000 tons were imported. It is expected that in the year 1957 the production will be nearly 2,000,000 tons, and in the following year 2,500,000 tons.

Lime Production in Rhodesia.

A new lime works has been opened at Colleen Bawn by Rhodesia Cement, Ltd. The vertical kiln is expected to produce 1,000 pockets of hydrated lime per day. Cement-lime mixtures are also to be produced.

Cement Production in France.

The production of cement in France in the year 1956 was 11,200,000 tons; this was an increase of 6 per cent. compared with the previous year.

Cement Production in Lebanon.

The production of cement in Lebanon in the year 1956 was 487,527 tons.

The Cement Industry in Peru.

Until the year 1955, when a new cement works was built near Lima, the only cement works in Peru was that near Lima of Compania Peruana de Cemento Portland S.A. Two other cement works are now nearing completion in northern Peru, another is being built in the central area of the country, a contract has been signed for the construction of another cement works in southern Peru, and there is a possibility of another works being built in the east and another in the south.

The Cement Industry in Madagascar.

An agreement has been made for the re-opening of the cement works at Amboania, which was closed six years ago. The estimated production is 150 tons a day.

An Electronographic Study of Silicate Hydrates.

THE following is abstracted from a report entitled " An Electronographic Study of the Morphology and Crystallization Properties of Calcium Silicate Hydrates," by A. Grudemo (Proceedings No. 26 of the Swedish Cement and Concrete Research Institute. Price Kr. 15).

It is generally accepted that among the various compounds formed during the reaction between Portland cement and water the calcium silicate hydrates play the dominating part in determining the physical properties and the development of mechanical strength during the setting of the cement gel. The nature of the cementing action would be better understood if detailed knowledge were available about the internal microstructure of the calcium silicate hydrates. Since, however,

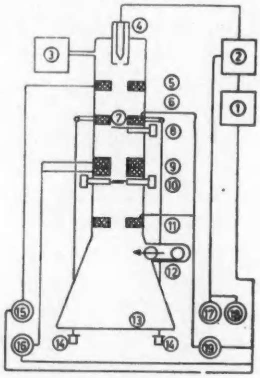

Fig. 1.
(1) Power supply and electronic equipment. (2) High-voltage transformer and rectifier. (3) Vacuum equipment. (4) Electron gun. (5) Condenser lens. (6) Objective lens. (7) Specimen probe and air-lock. (8) Exchangeable objective apertures. (9) Intermediate and diffraction lenses. (10) Diffraction aperture. (11) Projector lens. (12) Camera. (13) Fluorescent screen. (14) Levers for specimen movement. (15) Condenser control. (16) Magnification control. (17) Electron-beam intensity control. (18) High-voltage selector. (19) Focus control.

the crystals of these compounds are of submicroscopic or colloidal dimensions, ordinary light microscopy is generally inadequate. It might be assumed that size, shape, and aggregation properties of the solid particles are important factors in the formation of gel. The most suitable experimental method capable of providing new information about these properties seems to be electron microscopy, preferably in combination with electron diffraction. The studies described in this paper were started in 1950, since when a considerable amount of experimental material has been collected.

A three-stage Phillip's microscope (*Fig.* 1) was used. The lenses and controls are arranged so that the objective and projector lenses represent a fixed magnification (about 6,000 on the fluorescent screen, four times less on the photographic microfilm record), while the intermediate lens and the diffraction lens are used alternatively to change the magnification continuously from the fixed value up to about 80,000 times (the intermediate lens) or down to zero (the diffraction lens),

the last value representing the position of the electron diffraction. The most frequently-employed magnification for the photographic film is 5,000. As the resolution of the film is about ten times that of the human eye, the micrographs may subsequently be enlarged on positive prints up to ten times without appreciable loss in sharpness.

If an object is placed in the normal position (near the first focal plane of the objective lens) a diffraction diagram is formed as a "primary image" in the second focal plane of the objective. Although the diffraction image is very small, with a diameter of about 1 mm., most of its peripheral parts are cut off by the small objective aperture used to obtain good contrast in a normal microscope. If this aperture is changed for one of much larger diameter, an enlarged image of the diffraction diagram can be projected on to the screen by the combined action of the diffraction and projector lenses, with the diffraction lens current in the zero position.

The materials examined were obtained from various sources and by different methods. Some were obtained by mixing lime, silica, and water during ultrasonic treatment, or by hydration of C_3S by means of ultrasonic vibration or shaking for a long period. Other materials were obtained by adding ethyl orthosilicate to lime solutions, or by the slow hydrolysis of an ethyl orthosilicate layer on the surface of a solution of lime. Other reference materials and clay minerals were included for a comparison with the possible crystal habits and distortions of silicates with layer lattices.

The conclusions reached were as follows. Generally the various C—S—H products studied showed a considerable diversity of shapes and sizes of crystals, homogeneity, aggregation, etc., and no two products seemed to be exactly alike in these respects. It was observed that the most commonly-appearing low-temperature C—S—H compound, namely, CSH(B), may in favourable circumstances develop large crystal layers, although superposition of layers seems to be less common than in clays on account of the greater flexibility and ease of distortion of the layers. In saturated and supersaturated solutions of lime, however, a fibrous or needle-like growth of crystals seems to be promoted, probably caused by degeneration of the sheets into lath-like structures and by twisting or rolling of the crystal sheets. It is likely that aggregates of such structural elements are common in a setting cement gel, where they probably play a major part in determining the mechanical properties of the paste. However, as numerous examples show, the aggregation and interlacing of fibrous particles, as well as the interaction between fibres and other types of particles present, may proceed in many different ways depending on the conditions. In practice, the crystallisation processes are further complicated by the presence of other constituents, such as aluminate, ferrite, alkali, and sulphate ions, which may influence the crystalline properties and the microstructure of the C—S—H compounds.

The results of this investigation generally agree with the commonly accepted ideas of the morphological structure of cement gel as described by Professor J. D. Bernal at the Third International Symposium on Cement held in London in 1952.

High-Alumina Cement.

Patent Application No. 747,016, in the name of Messrs. Albright & Wilson Ltd., relates to a process for producing a high-alumina material for grinding to a cement. This process comprises reducing a phosphorous containing mineral mixture to form phosphorous and a slag containing aluminium, calcium, and silicon wherein the ratio alumina to calcia is between 0·5 and 1·85 and silica is up to 10 per cent. and contains less than 3·5 per cent. fluorine when the silica is less than 5 per cent., or less than 2·5 per cent. fluorine when the silica is 5 to 10 per cent. The slag is retained in a molten and quiescent state for a time sufficient to allow separation of iron phosphide. The mineral mixture may be a naturally occurring *a*-aluminium phosphate of low fluorine content, or *b*-aluminium calcium phosphate, or bauxite or alumina together with a naturally occuring calcium phosphate or defluorinated fluorapatite and bauxite.

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